

## IX. WELDABILITY OF TUNGSTEN BASE ALLOYS\*

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### SUMMARY

The weldability of unalloyed tungsten and two tungsten alloys was evaluated. The alloys were W-25Re (wt %) and W-25Re-30Mo (at. %). These were evaluated as arc cast material and for the ternary alloy also as a powder metallurgy product. The most important aspect of welding unalloyed tungsten was that it is very sensitive to thermal shock. High preheat temperatures (to 1400° F) alleviated this problem. W-25Re had improved thermal shock resistance but was shown to be basically hot tear sensitive in gas tungsten arc welding. Preheat temperatures of 1400° F were again beneficial. The powder metallurgy W-25Re-30Mo alloy displayed excellent weldability, whereas the arc cast material displayed extensive hot tearing and, hence, poor weldability. The anomalous hot tearing behavior of the arc cast W-25Re-30Mo alloy was ascribed to a very high sensitivity to oxygen contamination. The effects of postweld annealing, joint preparation, and aging for 1000 hours at temperatures to 3000° F were evaluated. The high temperature tensile strength of base metal, gas tungsten arc welds, and electron beam welds in W-25Re-30Mo was determined.

### INTRODUCTION

The studies described in this paper complement a series of programs designed to upgrade refractory metal alloy technology in terms of space power requirements. Contemplated systems would provide either direct conversion of thermal to electric energy as with thermoelectric or thermionic devices or mechanical conversion using Rankine or Brayton cycles. The major design objective of high thermal efficiency with minimum system weight is approached by designing for maximum operating temperatures. Application of tungsten or tungsten alloys seems to offer the ultimate potential in this respect because tungsten has the highest melting point of

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all metals, 6170<sup>0</sup> F. On the negative side, tungsten has a ductile-to-brittle transition temperature which is well above room temperature for recrystallized or cast (weld) structures. Hence, considerable reserve must be exercised in the application of this metal in fabricated structures typical of those required for space power systems.

This weldability study was designed to lend further definition to the general problems which would be encountered in fabrication of tungsten, or tungsten alloy structures, by welding. Stimulus for this evaluation was provided by the introduction of alloys of improved ductility such as the binary W-Re or ternary W-Re-Mo alloys. Further, techniques to convert these alloys from arc cast ingots have been recently developed. Arc cast material has historically demonstrated greater fabricability than the powder metallurgy product. Hence, the availability of arc cast material provided an additional incentive for initiating this welding study.

The basic objective of this program was to define the weldability of tungsten and its alloys in terms comparable to those employed in evaluating other refractory metal alloys (Cb or Ta based) which are prime candidates for space power system applications (ref. 1). The alloys of current interest in this respect are W-25Re (wt %) and W-25Re-30Mo (at. %). These were evaluated for the first time in this program as material converted from arc cast ingots along with arc cast unalloyed tungsten. The ternary alloy was also evaluated as a powder metallurgy product. The primary factors evaluated were

- (1) Basic weldability of sheet material using the gas tungsten arc and electron beam processes
- (2) Effect of weld atmosphere control on basic weldability
- (3) Effect of weld preheat to 1400<sup>0</sup> F
- (4) Importance of joint preparation
- (5) Effect of postweld annealing
- (6) Effect of long-time, high-temperature thermal exposure

## TECHNICAL PROGRAM

### Alloys Evaluated

The unalloyed tungsten and the tungsten alloys evaluated in this program are listed in the following table along with their respective melting points and densities.

	Melting point, °F	Density, lb/in. <sup>3</sup>
Unalloyed tungsten	6170	0.697
W-25Re (wt %)	5650	.714
W-25Re-30Mo (at. %) <sup>a</sup>	5270	.651

<sup>a</sup>Conventional designation of this alloy is in at. % and is used in this paper; composition in wt % is W-29.5Re-18.2Mo.

The unalloyed tungsten and the binary tungsten-rhenium alloy were evaluated solely as arc-cast (AC) sheet, while the ternary tungsten-rhenium-molybdenum alloy was evaluated both as arc-cast (AC) and powder-metallurgy (PM) sheet. Evaluation of arc cast material was emphasized because initial welding results on unalloyed tungsten showed that porosity free welds could only be made in arc cast material. Further, the general trend in refractory metal technology has historically been towards arc cast material for higher purity and greater fabricability.

The phase diagrams pertinent to these alloys are shown in figures IX-1, IX-2, and IX-3. In figure IX-3 the 1830° F (1000° C) isotherm for the W-Re-Mo ternary is shown. The location of the alloy composition used in this study is indicated. From these diagrams it is seen that both the binary and ternary alloys are nominally single phase but lie quite near the limiting solvus lines.

The binary W-Re and Mo-Re diagrams are quite similar. From the standpoint of weldability, however, a very important difference exists. W-Re alloys with compositions in the  $\alpha$ -phase region would be expected to be subject to considerably more constitutional segregation than would similar Mo-Re alloys. This follows from a direct comparison of the temperature range through which the metal must cool as it solidifies. Freezing point depression of the binary W-Re alloy would be expected to be pronounced in rapidly solidified cored weld structures. These phase relations imply that the W-Re-Mo system should experience considerably less segregation than the binary W-25Re alloy. This is based on the very narrow liquidus-solidus separation in the binary Mo-Re alloy for the ternary solute ratio (~60%Re). Data presented later in this paper tend to substantiate this expectation.

The interest in the binary W-Re alloy results from the well-known but poorly understood "rhenium ductilizing effect." This effect is not limited to W but has also been seen for Re additions to the other Group VIA metals, molybdenum and chromium. A recent review of this effect by Klopp (ref. 2) indicates the general lack of understanding of the mechanism(s) involved. Based on experimental evi-

dence two conclusions seem indicated:

(1) Re additions to Group VIA metals such as tungsten promote twinning as a major means of deformation. This implies a significant reduction in the normally high stacking fault energy of these metals.

(2) Some change in the morphology and/or distribution of interstitial compounds, particularly oxides, occurs. This would appear to be important since Stephens (ref. 3) has shown that the DBTT for pure W rises rapidly with oxygen content, the fractures being invariably intergranular.

The ternary W-Re-Mo alloy is a more recently developed material (ref. 4). Molybdenum additions to the W-Re binary alloys are attractive for several reasons. The ternary, with molybdenum replacing tungsten, is less expensive to produce and has a lower density than either W or W-Re binary alloys. However, the melting point is considerably lower and as a result the long-time high temperature strength is somewhat less than that of the higher melting binary alloys.

The short time strength properties determined for the ternary alloy are compared with typical values for arc cast tungsten and W-25Re in figures IX-4 and IX-5. Data relating the corresponding tensile elongations are listed in table IX-1. Up to 3000° F, the highest test temperature used, the differences are not very significant; but for higher temperatures, it is expected the ternary alloy would not continue to be competitive with the higher melting W-25Re and unalloyed tungsten.

Bend ductility (4t bend radius) of the as-received alloys is shown in table IX-2 along with notes regarding the as-received structures. Interstitial chemical analyses are provided in table IX-3. It is important to note that all of these metals have quite low solid solubilities for the interstitial elements. Hence, segregation of interstitials often occurs at grain boundaries and other regions of high disregistry in the lattice. This resultant segregation is thought to be responsible, in part, for the characteristic grain-boundary nucleated fractures so prevalent in these materials.

An unambiguous definition of the factors which control brittleness in tungsten and its alloys has not been achieved. However, it is well known that wrought, stress-relieved structures possess significantly greater ductility than that of recrystallized structures. This advantage has led to the widespread use of tungsten-base materials in the wrought, stress-relieved condition. This is the reason the materials used in this study were stress relieved rather than recrystallized. The influence of structure on ductility adds importance to the aging studies, which were conducted to assess the effects of long time, high temperature thermal exposures on structural stability.

An interesting feature of the interstitial analyses of table IX-3 is that, for the ternary alloy, the oxygen and nitrogen contents of the PM product are lower than those of the AC product. This is contrary to the normal relation and reflects the

fact that this alloy was originally developed as a PM product and evolved from a program which had as one of its major goals the development of techniques for obtaining extremely low interstitial impurity levels in tungsten and molybdenum alloy powders. The data in table IX-3 attest to the efficiency of these procedures. A similar comparison was not made for metallic impurities, but it is expected these would be somewhat lower in the AC sheet by virtue of the purification which occurs during vacuum arc melting.

## Alloy Weldability

Basic considerations. - Weldability of tungsten and tungsten alloy sheet was investigated by evaluating responses to electron beam and gas tungsten arc welding over a wide parameter range. This approach provides a delineation of alloy sensitivity to process variations and a definition of weldability limitations.

The primary welding variable in this respect is welding speed. Weld speed is the controlling factor in unit weld length heat input for achieving a given target weld size, as shown graphically in figure IX-6. The significant effect of weld speed is obvious in this figure. Heat input is nearly a function of  $1/v$ , or the dwell time of the arc. At slower speeds a small decrease in speed causes a large increase in heat input, and consequently increases the magnitude of the thermal disturbance. This effect would seem to be most important from a metallurgical standpoint. On the other hand, higher weld speeds can be considered to represent a greater thermal shock. In some materials the magnitude of the thermal disturbance plays the most significant role in establishing weldability limitations, while in others thermal shock is the overriding consideration. Because of the brittle nature of the materials evaluated in this program, thermal shock played a more important role in defining weldability.

Electron beam welding provides a minimum sized weld and hence minimum heat input throughout the welding speed range. This is also shown in figure IX-6. Frequently, minimizing weld size is beneficial in improving weld properties, but like higher speed GTA welding, minimizing heat input characteristically increases thermal shock. Again, this proved to be important in welding tungsten alloys, as described later in this paper. Hence, by employing both the GTA and EB welding processes in this study, extremes of both the thermal disturbance and thermal shock effects of welding were evaluated.

A further interesting feature of the heat input requirements developed in this program is the decrease in heat input for the higher solute content alloys (also lower melting point alloys). Hence, while the advanced tungsten alloys were devel-

oped for improved ductility alone, from a welding standpoint both improved ductility and lower thermal shock can potentially combine in these alloys for improved weldability. Decreased thermal shock in the alloys results from the lower heat input requirement (at a given weld size and welding speed) coupled with the lowered melting point. This combination decreases the instantaneous thermal gradient during welding.

Welding procedures. - All gas tungsten arc welding was conducted in a very pure, precisely controlled, helium environment employing the vacuum purged weld chamber shown in figure IX-7. The welding atmosphere was monitored during welding so that oxygen and moisture levels were always maintained at less than 5 ppm. The method of achieving and maintaining these purity levels is described in detail in references 1 and 5. During this investigation the importance of providing a high quality welding atmosphere for welding tungsten alloys was demonstrated. This aspect is discussed under the heading Hot Tearing in the RESULTS section of this paper. All gas tungsten arc welding was accomplished using straight polarity DC current.

Electron beam welding was accomplished using a Hamilton Zeiss 2-kilowatt, 150 000-volt welder. A vacuum of  $10^{-5}$  torr or less was employed for welding. Basic process variables evaluated included selected beam deflection patterns and clamp spacing as well as welding speed.

Either butt welds or bead-on-plate welds were used in this study. Geometric effects in welding narrow specimens dictated that most of the welds produced in this evaluation be bead-on-plate welds to conserve material. Hence, results in the weld evaluation are largely independent of joint preparation. However, the effect of joint preparation on the soundness of welds was separately evaluated.

Weld preheat. - As described previously, the general philosophy pursued in welding these alloys was that of treating the welding process as a thermal disturbance, the time-temperature relations of which are controlled by the weld parameter selection. Thermal shock proved to play a significant role in defining weldability of tungsten and its alloys. Consequently, weld preheat up to  $1400^{\circ}$  F was introduced as a variable into the welding study. Since  $1400^{\circ}$  F appeared to be above the ductile-to-brittle transition temperatures of both base and weld metal, preheat was selected as a means of providing increased flexibility in weld parameter selection. The preheat fixture designed for this purpose is shown in figure IX-8. This fixture was designed for sheet welding. The weld specimen is held in place with clamp down bars containing molybdenum inserts. The backup bar is also of molybdenum. The fixture heater is located in a cavity behind the molybdenum backup bar. Clamp bars, backup bar, and heater support are insulated from the bulk of the heater so that a maximum specimen temperature of  $1500^{\circ}$  F can be achieved.



Postweld annealing. - Postweld annealing was evaluated as a means of improving ductility of welds for all the material evaluated. Annealing was accomplished in diffusion pumped vacuum furnaces at a vacuum of  $<5 \times 10^{-5}$  torr and temperatures between  $2500^{\circ}$  and  $3200^{\circ}$  F. Holding times of 1 hour were employed for all anneals.

Thermal stability. - The thermal stability of welds in both powder metallurgy and arc cast W-25Re-30Mo was determined by aging for 1000 hours in ultrahigh vacuum furnaces at temperatures of  $2600^{\circ}$ ,  $2800^{\circ}$ , and  $3000^{\circ}$  F. The sputter-ion pumped furnaces used for this purpose are shown in figure IX-9. These units are capable of maintaining  $<10^{-8}$  torr pressure at temperature. Pressures tend to continually decrease during aging runs such that final pressures are  $\sim 10^{-9}$  torr.

Weld evaluations. - All welds made in this program were checked for basic quality using visual, dye penetrant, and radiographic techniques.

The primary mechanical method of evaluation was bend testing using a 4t bend radius (11% outer fiber strain). Bend testing was used to define the bend-ductile-to-brittle transition temperature for weld specimens taken in both the transverse and longitudinal directions. The bend test parameters and specimen orientations are defined in figure IX-10. Transverse specimens were oriented for bending with the weld axis at a slight angle to the punch axis to ensure that the entire weld transverse cross section would be subjected to bending rather than merely the weakest areas. Load-deflection curves were generated during each bend test, and bending was terminated when crack initiation was indicated by an abrupt load decrease. This permits measuring, or calculating, the bend angle achieved at the moment of crack initiation as well as identifying the location of crack initiation. Normally four specimens are required to define a transition temperature. Bend test data are recorded graphically as shown in figure IX-11. This method of presentation identifies all the pertinent data, including crack location and extent of crack propagation for each specimen as well as the transition curve defined by the bend angle achieved as a function of temperature. Longitudinal and transverse curves are coded for presentation on the same graph. Bend testing was conducted at temperatures up to  $1000^{\circ}$  F, the test fixture operating limit. Some anomalous results were noted when the rhenium containing alloys were tested in air above  $600^{\circ}$  F. This was attributed to the tendency of rhenium to form low melting oxides, which demonstrated that an inert shield gas should be employed in bend testing these alloys. The expanded discussion of this general problem is included in the discussion of results under the heading Hot Tearing.

A restricted amount of tensile testing was conducted using longitudinal GTA and EB weld specimens and base metal of the W-Re-Mo alloy. These data are presented in figures IX-4 and IX-5. Tensile tests at elevated temperatures were conducted at strain rates of 0.05 inch per inch per minute while room temperature

tests were run at 0.005 inch per inch per minute to the 0.6 percent offset yield point and then at 0.05 inch per inch per minute to failure. Weld specimens were ground flat and parallel. A 1.000 inch long by 0.250 inch wide gage length was employed.

Specimen preparation. - The tungsten alloys did not lend themselves to convenient specimen blanking because of generally poor ductility. As a result, weldment specimen blanking throughout this program was accomplished by electrodischarge machining. Following welding, bend and tensile specimens were blanked using a wet cutoff wheel. Tensile specimens and butt joint edges were finish machined by grinding. All specimens were pickled before welding, annealing, aging, or testing above 1000<sup>0</sup> F. All other specimens (bend) were degreased prior to testing. Selection of the pickling procedures is discussed in the RESULTS section of this paper since proper pickling techniques are necessary to avoid excessive weld porosity.

## RESULTS

### Basic Weldability

Weld parameters, weld inspection results, and bend transition temperatures for all welds produced in screening the four materials for basic weldability are summarized in tables IX-4 and IX-5. All the variables investigated are indicated. Extreme care was taken to hold all other possible variables constant. This included electrode configuration, arc gap, shielding gas, edge preparation, and clamp spacing in GTA welding, and beam focus and voltage in EB welding.

Weld size was treated as a general variable in GTA welding, and target weld sizes were selected. Since any particular application would require a particular weld size, and since heat input is a function of weld size, size was considered an important metallurgical variable. In electron beam welding, however, weld size (width) is a much more independent variable which is usually held as small as possible. Hence, EB weld size was not treated as a practical variable. Clamp spacing was treated as a variable in EB welding but was held constant in GTA welding.

EB welding speeds were higher than those used for GTA welding, as is normal. Although higher weld speeds were attempted in GTA welding, the lower speeds were necessarily favored in an effort to increase the probability of obtaining sound welds. Hence, the indicated parameters reflect a chronological adjustment of the original plan which was sensibly altered as the evaluation proceeded.

The types of defects which occurred varied considerably for the four alloys:

(1) Arc cast unalloyed tungsten welds failed apparently as a result of brittleness and hence inability to accommodate weld stresses. EB welding produced the most

dramatic failures, which included delamination of adjacent base metal as well as transverse cracks and fractures (figs. IX-12 and IX-13). The EB delaminations are apparently the result of the high thermal shock developed in this welding process. High preheat (1400° F) improved GTA weldability, particularly as indicated by the ability to produce larger welds at higher speed. Weld fractures of the type indicated were the only types of defects detected in welding unalloyed tungsten.

(2) Arc cast W-25Re, like unalloyed tungsten, was GTA welded with difficulty. However, it was readily EB welded. GTA welding became increasingly difficult with higher welding speeds. Transverse arrested cracks (weld and heat affected zone only) occurred in one 15-inch-per-minute weld and in three 30-inch-per-minute welds. One 7.5-inch-per-minute weld contained a centerline crack which may have been a hot tear. Such cracks were also observed in welding a circular bead-on-plate patch test specimen in this alloy. The 1400° F preheat proved advantageous in this respect with only one short starting tear developing in a 15-inch-per-minute weld. There was no need to evaluate preheating for EB welding of this alloy because of the excellent weldability displayed.

(3) The powder metallurgy W-Re-Mo alloy displayed excellent weldability using both the GTA (fig. IX-14) and EB welding processes with only one minor starting crack occurring in one GTA weld.

(4) The arc cast W-Re-Mo behaved in a very anomalous manner by hot tearing (fig. IX-15) and developing transverse cracks during GTA welding. Although EB welding was satisfactory, this material was essentially unweldable by the GTA process. This was unexpected, and this problem was given special attention, as discussed later.

### Supplemental Weldability Results

The other important features of basic weldability evaluated in this program are discussed in this section. These included the effect of weld parameters on as-welded ductility, the effect of weld preheat, the effect of postweld annealing, a comparison of edge preparation methods (pickling solutions), and a comparison of porosity in arc cast and powder metallurgy W-Re-Mo alloys.

The effect of weld parameters on the ductility of welds as measured by the bend transition temperature is summarized as part of the basic weldability data in tables IX-4 and IX-5. Bend test results were carefully reviewed, but no correlation was established based on a thermal response analysis as previously accomplished using a similar approach for evaluating columbium base alloys (ref. 1). In this study failure to achieve a satisfactory correlation is ascribed to the nominal variability of

properties associated with the brittleness and/or hot tear sensitivity of these materials. From a statistical standpoint these materials can be expected to behave inconsistently. Hence, a much greater sample is required to achieve a meaningful correlation than required with readily weldable materials.

The variation of weld preheat, like the other weld parameters, was ineffective in demonstrating a definite trend in controlling as-welded ductility. However, as previously described, preheating was very instrumental in improving weldability (i. e., preheating enhanced flexibility in terms of insensitivity of weld quality to variation of the conventional welding parameters). This advantage was realized most effectively with the 1400° F preheat.

Preheating is not required for GTA welding W-25Re-30Mo if the welding characteristics of the powder metallurgy alloy can be consistently realized. On the other hand, not even preheat was beneficial in GTA welding arc cast W-25Re-30Mo.

Preheating is not beneficial for EB welding the tungsten alloys but is probably necessary for EB welding unalloyed tungsten and is preferred for GTA welding tungsten. Preheating for GTA welds in W-25Re is necessary only with high welding speeds.

The effect of postweld annealing as a method of improving as-welded ductility is summarized in table IX-6. Unalloyed tungsten was evaluated with a 1 hour, 2560° F GTA weld stress relief only without realizing any benefit. The same anneal on W-25Re was quite effective for EB welds but ineffectual for GTA welds. This was interpreted as indicating that a stress relief of EB welds is desirable. This also indicated that residual stresses are not the controlling factor in GTA weld ductility impairment. However, W-25Re GTA weld ductility was improved with a 3270° F anneal. This temperature was selected for solution of nonequilibrium sigma phase which could be responsible for ductility impairment. Even though sigma phase was not detected metallographically, its presence as a continuous or semicontinuous grain boundary or intercellular film in welds can be inferred from the intergranular nature of the fracture observed and from the improved ductility realized with the high temperature anneal.

Powder metallurgy W-Re-Mo welds were improved by annealing in the stress relief and potentially sigma forming range, 2400° and 2800° F, but not in the recrystallization-sigma solution range, near 3200° F. Hence, development of sigma phase did not appear to be a problem with this alloy. Arc cast W-Re-Mo, which had better as-welded ductility (EB welds only) than the powder metallurgy sheet, decreased in ductility on annealing to about the same final level as annealed powder metallurgy welds. Hence, these two materials merely seemed to normalize through the thermal stability study, as discussed later. Annealing naturally has a detrimental effect on wrought base metal, as indicated for the W-Re-Mo alloy an-

nealed at 2800° F.

Pickling solution selection proved to have a significant influence on the occurrence of porosity in the W-25Re-30Mo alloy. The developer of this alloy recommended using a volume ratio solution of 30 lactic acid - 3 HNO<sub>3</sub> - 1 HF. This was compared with the 9HF-1HNO<sub>3</sub> solution which was used satisfactorily for preparing the other two alloys. Specimens pickled with both solutions were degassed in vacuum (10<sup>-5</sup> torr) at 2000° F prior to welding, as was the practice throughout this program for all weld blanks. The results of this investigation are shown in figure IX-16. The recommended solution is clearly preferred for the W-25Re-30Mo alloy to avoid weld porosity even though the 9HF-1HNO<sub>3</sub> solution was satisfactory for the other materials.

Another factor resolved in this evaluation is the greater tendency of powder metallurgy alloy welds than arc cast alloy welds to contain porosity. Typical results in this respect are shown in figure IX-17. Powder metallurgy W-25Re-30Mo consistently displayed a greater tendency towards weld porosity than did the arc cast material.

The reason for this could not be determined, but the trend agrees with that observed in the preliminary survey for this program leading to the selection of arc cast rather than powder metallurgy tungsten for evaluation. No correlation between weld ductility and porosity was demonstrated. Several welds in W-25Re-30Mo were produced with high porosity and bend tested without any apparent increase in transition temperature.

## Hot Tearing

Hot tearing, quite often catastrophic in extent, occurred in gas-tungsten-arc welds in the AC W-25Re and the AC W-Re-Mo alloy with sufficient regularity to warrant closer examination in an effort to identify the causes. The problem was serious enough in the AC ternary alloy that full-scale evaluation of GTA welds was not possible because of a lack of sound weld metal.

Hot tearing is not peculiar to welds; rather it is a problem common to many aspects of metallurgical processing. Although a precise definition of the obtaining mechanisms has proven elusive, a definite relation has been established between the occurrence of hot tearing and the existence of a liquid phase at temperatures well below the solidus temperature of the alloy. This situation is often predictable from the equilibrium diagram (ref. 6). The inability of the liquid phase to accommodate strains induced by solidification and subsequent shrinkage results in parting at the liquid film region. At first appearance it might be expected EB welds would

be more subject to this problem than GTA welds because of their high cooling rates. However, the instantaneous volume of liquid present and magnitude of thermal straining is quite small for EB welds, and this apparently mitigates the tendency for hot tearing.

It was previously noted that a high degree of constitutional segregation and subsequent depression of the freezing point in weldments is expected in the W-Re system (fig. IX-1). This could play two possible roles in the hot tearing noted in W-Re binary alloy welds. Should the cooling rate be sufficiently great, the Re-rich phase, that is, the last constituent to solidify, could serve to fulfill the liquid film requirements outlined above and induce hot tearing. A more subtle role, also related to the existence of a Re-rich phase, stems from the high affinity which Re exhibits for oxygen. Although easily formed,  $\text{Re}_2\text{O}_7$  is unstable, melting at  $565^\circ\text{F}$  and boiling at  $685^\circ\text{F}$ , and is believed to be responsible for the hot shortness which prevents elevated temperature working of Re in air (ref. 7).

The low level of oxygen in the binary W-Re (table IX-3) and the ultraclean welding procedures followed seem to obviate consideration of the latter mechanism as being responsible for the observed hot tearing. However, this mechanism seems quite feasible as an explanation for the anomalous bend test results noted for tests in air at temperatures above  $\sim 600^\circ\text{F}$ . The use of an inert (argon) shield gas eliminated this erratic behavior.

As opposed to the binary alloy the hot tearing of GTA welds in the AC W-Re-Mo alloy was totally unexpected. First, the amount of constitutional segregation expected in this alloy is not nearly so great as that expected for the binary W-Re alloy. Hence, the possibility of a depressed-melting-point liquid film at a critical stage in the solidification is not as likely. Second, GTA welds of the PM W-Re-Mo alloy were accomplished without a single incident of hot tearing.

In an effort to identify the cause(s) for this dual behavior, a complete review was made of the processing histories and the chemical analyses of the AC and PM sheets. This review indicated differences in oxygen content (table IX-3) for the two products might be responsible for the erratic weldability of the AC sheet. The mechanism would be similar to that which has been observed in welding molybdenum. It has been reported (ref. 8) and verified (ref. 9) that oxygen contents of only 100 ppm (by wt.) in molybdenum have been sufficient to consistently lead to hot tearing during welding. This is related to the presence of a continuous film of Mo-MoO<sub>2</sub> eutectic (melting point  $\sim 3800^\circ\text{F}$ ) at the grain boundaries for oxygen concentrations of 100 ppm or more. Fractographic studies (ref. 10) indicate the transition from discrete oxide particles to a continuous grain boundary film may occur for oxygen levels as low as only 10 to 50 ppm. Accumulation of critical oxygen concentrations could conceivably result from partitioning effects between the solid and

liquid phases during solidification.

Evidence which tends to confirm that this mechanism is responsible for hot tearing of GTA welds in the AC W-Re-Mo sheet was obtained by inducing similar behavior in GTA welds in the PM sheet. Hot tearing had not been noted in the PM product, yet severe hot tearing was induced in a series of test welds made in oxygen-contaminated welding atmospheres. Photographs of two of these welds are shown in figure IX-18. Immediately below each weld is a positive print of an X-ray negative of the same weld. The threshold for the hot tearing occurred at approximately 500 ppm oxygen in the welding atmosphere. Attempts to more accurately define this behavior by chemical analyses for oxygen pickup in the weld metal met with limited success.

Although the oxygen effect hypothesis for this experiment was based on the effect of oxygen on hot tear sensitivity in molybdenum, the extension to alloys containing tungsten and rhenium seems reasonable because of their similarity in chemical behavior to molybdenum, particularly with respect to interstitial elements.

## Thermal Stability

The objective of the 1000 hour aging runs was to determine the effects of long-time high-temperature exposures on the ductility of the ternary W-Re-Mo alloy. Base metal and EB and GTA welds of the PM sheet were aged, while for the AC sheet only base metal and EB welds were used. All welds used in the aging study were made using the parameters found previously to give welds having optimum ductility. In addition, wherever material availability permitted, additional specimens were first aged and subsequently welded, again using optimized weld parameters.

For single phase alloys, such as the ternary W-Re-Mo alloys evaluated, the effects of long time exposures at elevated temperatures are mainly those associated with primary grain growth. In tungsten-base alloys this results in loss of ductility. The proximity of the alloy to the alpha-sigma phase boundary (fig. IX-3) suggests the possibility of an embrittling reaction due to localized precipitation of sigma phase during aging. To allow for this possibility, three sets of specimens were aged at 2800° F. One set was tested as aged, while the other sets were given 1 hour postage anneals at 3200° and 3400° F to dissolve any sigma phase that may have formed.

Bend test results pertinent to these efforts are summarized in table IX-7. Data for as-received PM and AC sheet and PM sheet annealed 1 hour at 2800° F are included to provide information regarding changes in ductility not related to welding.

The transition temperature for longitudinal (L) and transverse (T) test specimens are indicated as well as the average of these two values.

Ductility of the base metal specimens decreased with increasing thermal exposure. This was true for both the AC and the PM sheet over the full range of conditions evaluated. Metallographic examination was performed in an effort to determine the cause for this behavior. The results, shown in figure IX-19 (dashed lines) as recrystallized grain size as a function of temperature, indicate grain growth as the mechanism most likely responsible for the loss of ductility. Special attention should be directed toward the results found for the PM product. This alloy exhibited both normal and secondary grain growth for all aging temperatures, and hence two curves are shown for these specimens. The volume of the specimen affected by secondary recrystallization (i.e., abnormal grain growth) increased with aging temperature such that after 1000 hours at 3000° F only quite small areas remained unaffected. To provide additional information regarding this phenomenon, a series of 1 hour anneals at 200° F intervals from 2200° to 3600° F were given base metal specimens of the PM W-Re-Mo alloy. Specimens of the AC W-Re-Mo alloy and the AC W-25Re alloy were similarly annealed to provide direct comparisons of thermal response. These results are also included in figure IX-19 (solid lines), where the AC binary and ternary alloys are seen to observe normal grain growth behavior; that is, although the average grain size increases, the distribution of grain sizes remains nearly constant throughout the process. Again, secondary recrystallization was noted for the PM W-Re-Mo specimen annealed 1 hour at 3600° F (fig. IX-20).

Thermal exposure had no discernible effect on the bend ductility of EB and GTA welds in the PM sheet or on the ductility of EB welds in the AC sheet. This was found to be true for welds made by either sequence, weld and age or age and weld. In view of the complexity of responses possible for the variety of conditions employed, it is evident that the data lend themselves best to a rationale developed strictly on the basis of grain size.

The bend transition temperatures leveled off with increased thermal exposure. This suggests a lower limit of ductility is being approached for the W-Re-Mo alloy. Fractures in aged PM and AC specimens were invariably intergranular. Probably the greatest constitutional segregation coupled with minimum transverse grain boundary length occurs at the weld centerline. These factors probably combine and result in high transverse transition temperatures, since transverse specimens almost always failed along the weld centerline grain boundaries.



## CONCLUSIONS

1. The weldability of unalloyed tungsten is marginal because of its high ductile-to-brittle transition temperature in the welded or recrystallized condition. The high melting point and low ductility in combination make tungsten susceptible to failure by thermal shock during welding. Hence, weldability is enhanced by high weld preheating. It is not apparent that use of arc cast tungsten is advantageous over powder metallurgy tungsten except for the absence of porosity in welds. Post-weld annealing was not particularly beneficial in improving ductility.

2. The weldability of W-25Re is improved over that of unalloyed tungsten because of slightly better as-welded and recrystallized ductility. Improved ductility coupled with a lower melting point makes this alloy less susceptible to thermal shock failures. However, the W-Re phase relations are such that this alloy exhibits a tendency toward hot tearing.

Preheat in welding was not beneficial in improving as-welded ductility but permitted welding at higher welding speeds and, hence, essentially improved weldability.

A stress relief postweld anneal (2560° F) was beneficial for EB welds. This implied high residual stress in EB welded W-25Re tends to correlate with the thermal shock behavior observed for tungsten EB welds. GTA welds were not improved by stress relief, but instead required a solution anneal (3270° F), which implies that sigma phase develops at grain boundaries during GTA welding. In this respect EB welding was advantageous since embrittlement by the sigma phase and hot tearing were observed only in GTA welds. Both the development of sigma phase and hot tearing result from constitutional segregation on freezing which is apparently more pronounced in GTA welds.

3. The W-25Re-30Mo alloy displayed generally excellent weldability except for an extreme sensitivity to oxygen contamination which causes hot tearing. Undesirable levels of oxygen contamination occur at a very low level in the base metal and make detection difficult. Welding atmospheres, however, can be easily controlled if properly monitored to eliminate welding as a potential source of contamination.

A postweld stress relief was beneficial in improving the ductility of welds in this alloy. Otherwise, all thermal treatments to which this material was exposed tended to normalize ductility to that of a large grain size recrystallized structure. This trend persisted even through 1000 hour anneals at temperatures to 3000° F.

On aging this alloy tends to behave quite simply as a solid solution system. However, the powder metallurgy material exhibited secondary recrystallization which could well be a metallurgical instability brought on by the dissolution of finely dispersed impurity precipitates.

4. In several checks made in this program welds in powder metallurgy product always contained porosity, whereas arc cast material produced porosity-free welds.

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TABLE IX-1. - TENSILE ELONGATION DATA FOR

## TUNGSTEN-BASE ALLOYS

Alloy	Temperature, °F		
	2000	2600	3000
	Elongation in 1-in. gage length, %		
AC Tungsten	12	23	30
AC W-25Re (wt %)	5	16	30
PM W-25Re-30Mo (at. %)			
Base <sup>a</sup>	54	36	33
EB	33	26.5	25
GTA	37	23.5	18
AC W-25Re-30Mo (at. %)			
Base <sup>a</sup>	9	32	79
EB	15	17	61

<sup>a</sup>Wrought, stress-relieved sheet.

TABLE IX-2. - BASE METAL BEND DUCTILITY

Alloy	Longitudinal	Transverse	As-received condition
	Bend transition temperature for 4t rad., °F		(a)
Tungsten	425	275	Stress relieved 1 hr at 1700° F
W-25Re (wt %)	-200	-75	Stress relieved 1 hr at 2550° F
PM W-25Re-30Mo (at. %)	-150	-50	Stress relieved 1/2 hr at 2100° F
AC W-25Re-30Mo (at. %)	<-320	-250	Stress relieved 1/2 hr at 1920° F

<sup>a</sup>All as-received structures were in wrought condition.

TABLE IX-3. - BASE METAL INTERSTITIAL CHEMICAL ANALYSES

Alloy	Carbon		Oxygen		Nitrogen	
	ppm (wt.)	ppm (at.)	ppm (wt.)	ppm (at.)	ppm (wt.)	ppm (at.)
AC Tungsten	<sup>a</sup> 8	<sup>a</sup> 122	<sup>a</sup> 12	<sup>a</sup> 138	10	131
AC W-25Re (wt %)	8	123	8	92	10	132
AC W-25Re-30Mo (at. %)	48	632	24	237	10	113
PM W-25Re-30Mo (at. %)						
Lot A	<sup>a</sup> 19	<sup>a</sup> 250	<sup>a</sup> 5	<sup>a</sup> 49	<3	<34
Lot B	<sup>a</sup> 81	<sup>a</sup> 1070	<sup>a</sup> 4	<sup>a</sup> 39	<3	<34

<sup>a</sup>Average of two analyses.

TABLE IX-4. - GTA WELD PARAMETER EVALUATION

Target weld size	Weld speed, in. /min	Arc cast unalloyed tungsten			Arc cast W-25Re (wt %)			W-25Re-30Mo (at. %)					
		No pre-heat	450 <sup>o</sup> to 600 <sup>o</sup> F preheat	1400 <sup>o</sup> F preheat	No pre-heat	450 <sup>o</sup> to 600 <sup>o</sup> F preheat	1400 <sup>o</sup> F preheat	Powder metallurgy			Arc cast		
								No pre-heat	800 <sup>o</sup> F preheat	1400 <sup>o</sup> F preheat	No pre-heat	800 <sup>o</sup> F preheat	1400 <sup>o</sup> F preheat
4t bend transition temperature, <sup>o</sup> F (a), (b)													
Small welds, 0.120" wide, nominal (0.080 to 0.140")	3.0	-----	-----	-----	1000, L, S	800, L, S	-----	-----	-----	-----	---	-----	-----
	7.5	D	>1000, L, S	>1000, L, T, S	800, L, S	590, L, S	-----	-----	-----	-----	---	-----	-----
	15	S	1000, L, S	850, L, 800, T, S	600, L, S	-----	1000, T, D	400, L, S	500, L, S	-----	D	450, L, T, S, D, D	250, L, D, D
	25	-----	-----	-----	-----	-----	-----	-----	550, L, S	450, L, S	---	-----	-----
	30	700, L, D	-----	-----	-----	>1000, L, D	>1000, L, T, S, S	500, L, S, S	-----	-----	D	450, L, 350, T, S	-----
	35	-----	-----	-----	-----	-----	-----	-----	400, L, S	550, L, S	---	-----	-----
	45	-----	-----	-----	-----	-----	800, L, T, S	-----	-----	-----	---	-----	-----
Large welds, 0.180" wide, nominal (0.150 to 0.210")	3.0	-----	-----	-----	-----	-----	-----	-----	-----	-----	---	-----	-----
	7.5	700, L, S	700, L, S	-----	1000, L, S	D	-----	-----	-----	-----	---	-----	-----
	15	D	S	-----	1000, L, D	>1000, L, S	-----	-----	-----	450, L, D	---	-----	-----
	25	-----	-----	-----	-----	-----	-----	-----	-----	-----	---	-----	-----
	30	-----	D	800, L, T, S, S	D	>1000, L, D	-----	-----	-----	-----	---	-----	-----
	35	-----	-----	-----	-----	-----	-----	-----	-----	-----	---	-----	-----
	45	-----	-----	-----	-----	-----	-----	-----	-----	-----	---	-----	-----

<sup>a</sup>Longitudinal bend, L; transverse bend, T.<sup>b</sup>Sound weld, S; defective weld, D.

TABLE IX-5. - EB WELD PARAMETER EVALUATION

Weld speed, in. /min	Direction of 60-cycle, 0.050 in. beam deflection	Clamp spacing, in.									
		3/16	1/2	3/32	1/4	3/8			3/16	3/8	
		Arc cast alloyed tungsten; no preheat	Arc cast W-25Re (wt %); no preheat	W-25Re-30Mo (at. %)							
				Powder metallurgy			Arc cast				
				No preheat	800 <sup>o</sup> F preheat	1400 <sup>o</sup> F preheat	No preheat	1400 <sup>o</sup> F preheat			
4t bend transition temperature, <sup>o</sup> F (a), (b)											
15	Transverse	D	<1000, L, D	-----	400, L, >1000, T, S	-----	-----	-----	-----	-----	
	Zero	-----	-----	-----	400, L, >1000, T, S	-----	-----	-----	-----	-----	
	Longitudinal	<800, L, >1000, T, D	>1000, L, T, D	600, L, >1000, T, S	500, L, >1000, T, S, S	200, L, 600, T, S	-----	150, L, 550, T, S	-----	-----	
25	Longitudinal	<800, L, >1000, T, D	>1000, L, T, D	450, L, >1000, T, S	-----	200, L, 500, T, S, S, S	275, L, 500, T, S	150, L, 600, T, S	150, L, 250, T, S	175, L, 150, T, S	
50	Zero	-----	-----	>1000, L, T, S	400, L, >1000, T, S	-----	-----	-----	-----	-----	
	Longitudinal	D	D	600, L, >1000, T, S	600, L, T, S	225, L, 500, T, S	200, L, 600, T, S	250, L, 600, T, S	150, L, 250, T, S	150, L, 200, T, S	
100	Zero	D	D	-----	-----	-----	-----	-----	-----	-----	
	Longitudinal	D	D	900, L, >1000, T, S	>1000, L, T, S	-----	-----	-----	-----	-----	

<sup>a</sup> Longitudinal bend, L; transverse bend, T.<sup>b</sup> Sound weld, S; defective weld, D.

TABLE IX-6. - POSTWELD ANNEALING RESULTS

Alloy	Structure	Weld preheat	1 hr anneal temper- ature, °F	Change in 4t bend transition temperature, °F (a)	Bend type (b)	Lowest DBTT, °F (b)
W	GTA weld	None	2560	100	L	700
W	GTA weld	550° F	2560	-100	L	900
W	GTA weld	None	2560	Increased	L	700
W-25Re	GTA weld	550° F	2560	200	L	800
W-25Re	4 GTA welds	3-550° F 1-None	2560	Increased ductility implied	-----	800
W-25Re	4 GTA welds	1-550° F 3-None	2560	Decreased ductility implied	-----	800
W-25Re	3 GTA welds	1400° F	3270	-400 Max.	L and T	600
W-25Re	GTA weld	1400° F	3270	Questionable	-----	1000
W-25Re	11 EB welds	None	2560	-500 Max.	T	500
W-Re-Mo (PM)	GTA weld	None	2400 2800	-50 to -100	L	350
W-Re-Mo (PM)	GTA weld	None	3200	25	L	425
W-Re-Mo (PM)	EB welds	None	2400 2800 3200	-25 >-25 25	} L and T	175 (L)
						400 (T)
W-Re-Mo (PM)	Base metal	-----	2800	125	L	-150
W-Re-Mo (AC)	EB welds	1400° F	2400	175	T	-75
				50	L	150 (L)
				200	T	
			2800	50	L	200 (T)
				250	T	
			3200	100	L	
				250	T	

<sup>a</sup>DBTT for annealed or unannealed, whichever is lower.<sup>b</sup>Longitudinal bend, L; transverse bend, T.

TABLE IX-7. - SUMMARY OF BEND TEST RESULTS PERTINENT TO THERMAL STABILITY

[illegible]<sup>a</sup>Bend type, longitudinal, L; transverse, T.

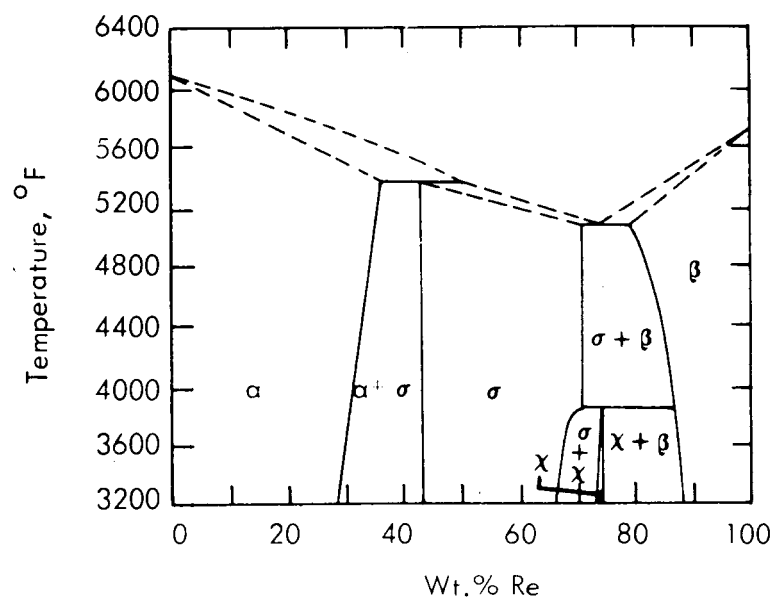


Figure IX-1. - Tungsten-rhenium phase diagram (ref. 11).

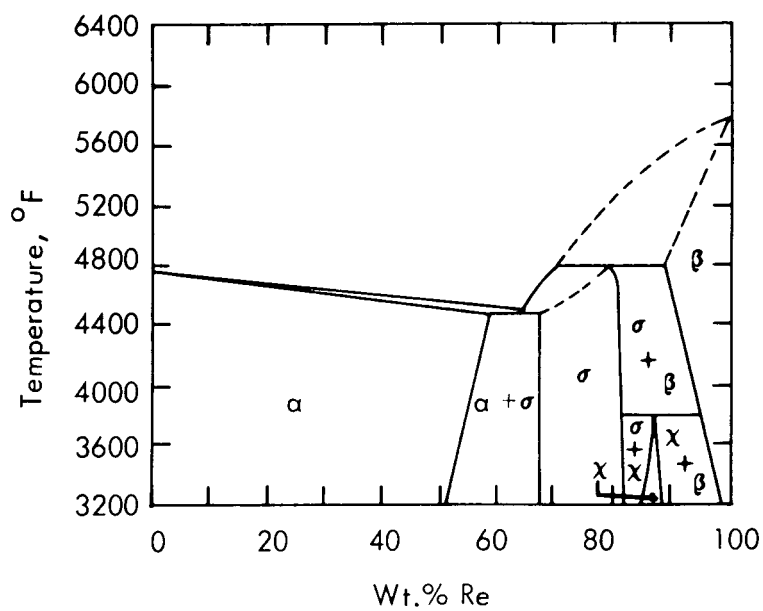


Figure IX-2. - Molybdenum-rhenium phase diagram (ref. 12)



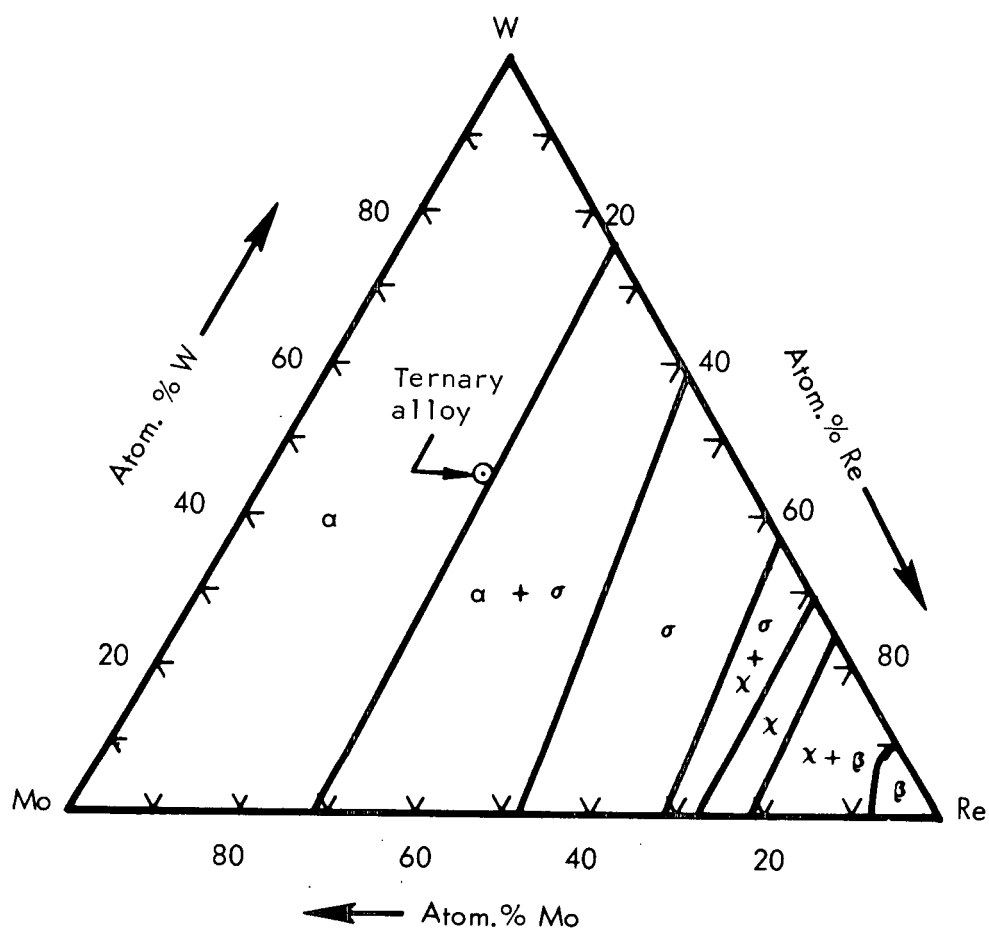


Figure IX-3. - Tungsten-rhenium-molybdenum ternary phase diagram: 1830<sup>0</sup> F isotherm (ref. 4).

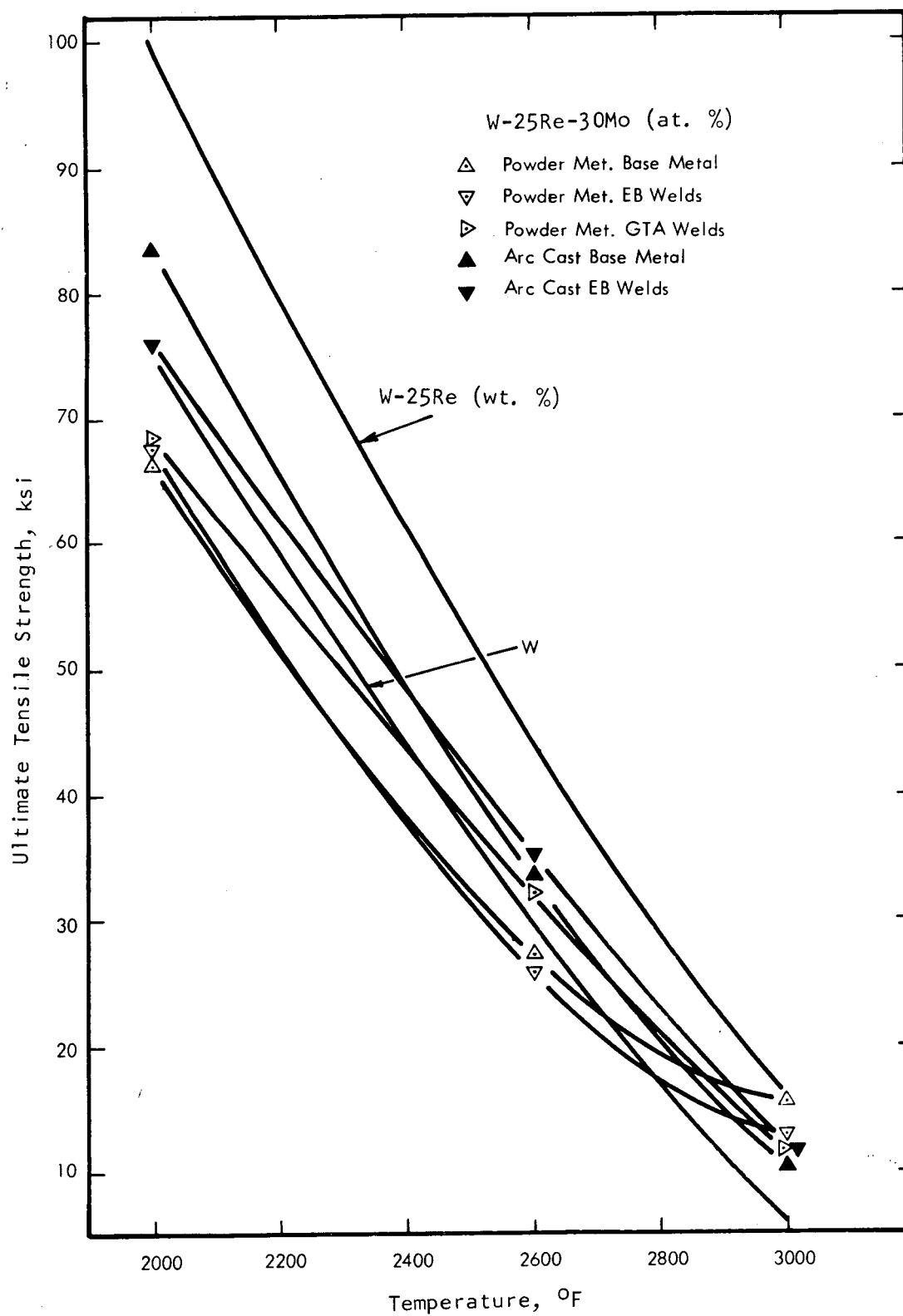


Figure IX-4. - Elevated temperature ultimate tensile strength of tungsten-base alloys.

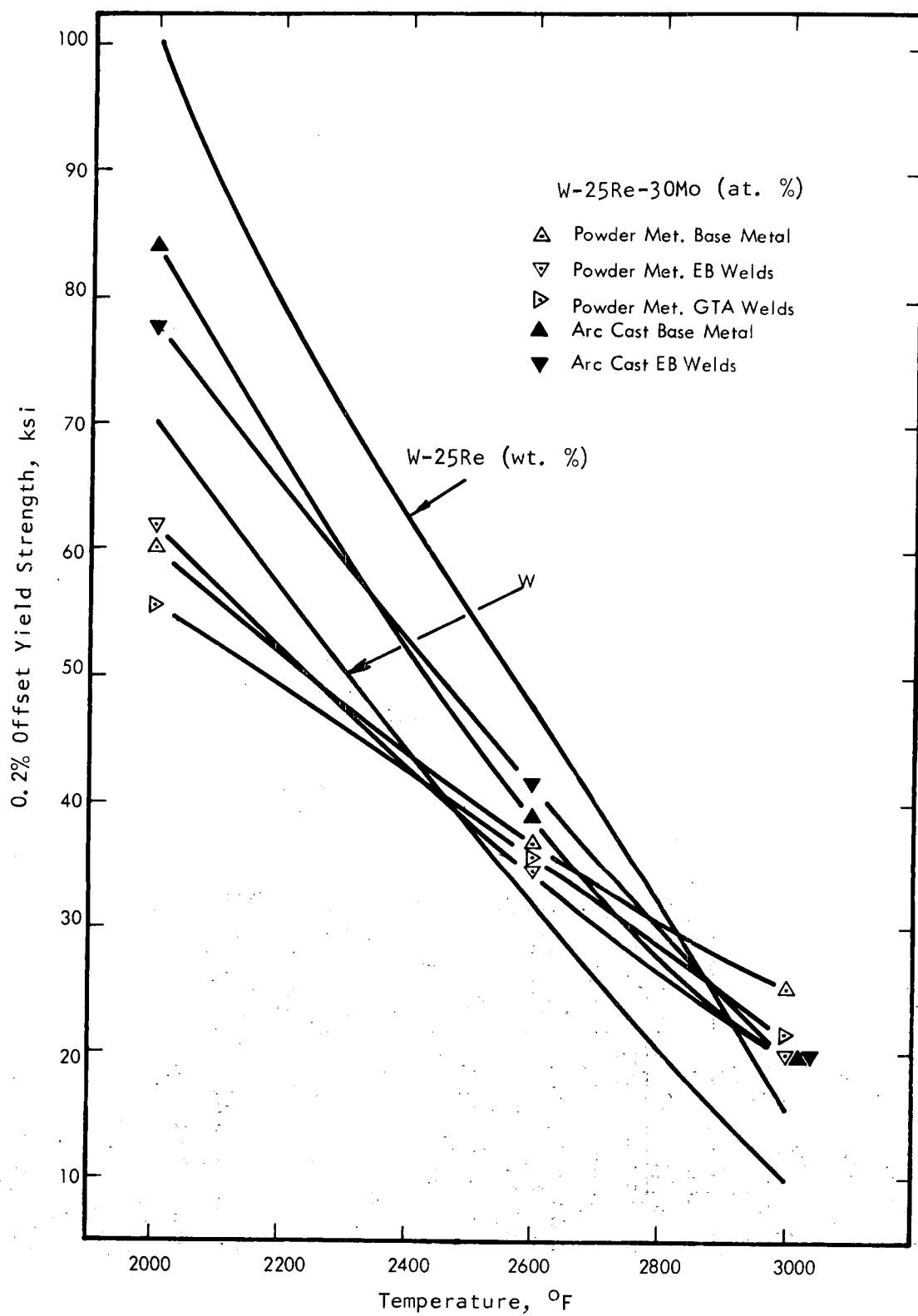


Figure IX-5. - Elevated temperature 0.2 percent offset yield strength of tungsten-base alloys.

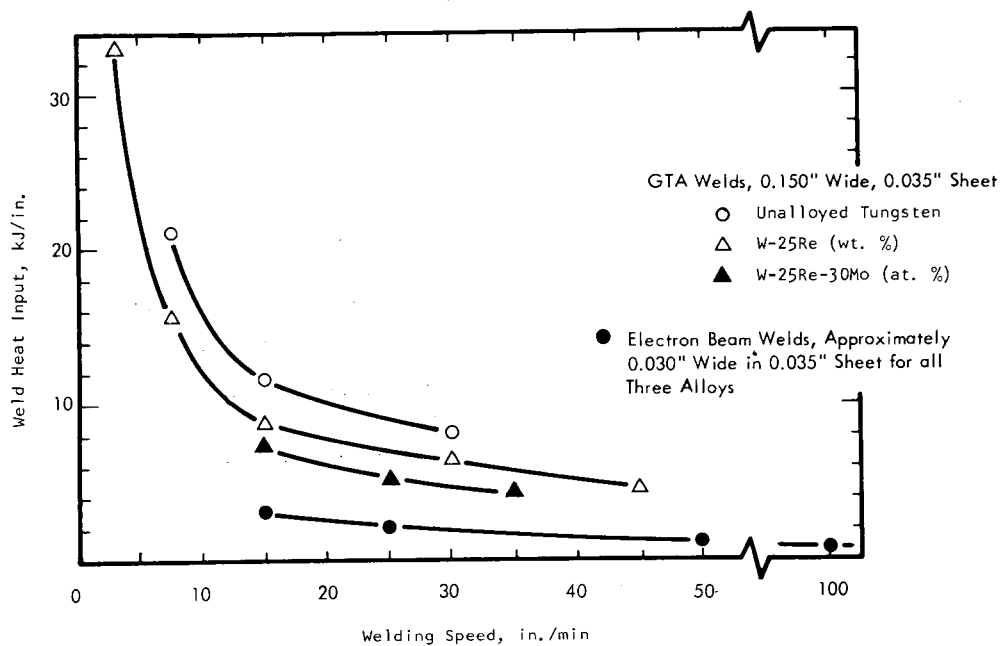


Figure IX-6. - Unit weld length heat input requirements as function of welding speed for tungsten-base alloys.

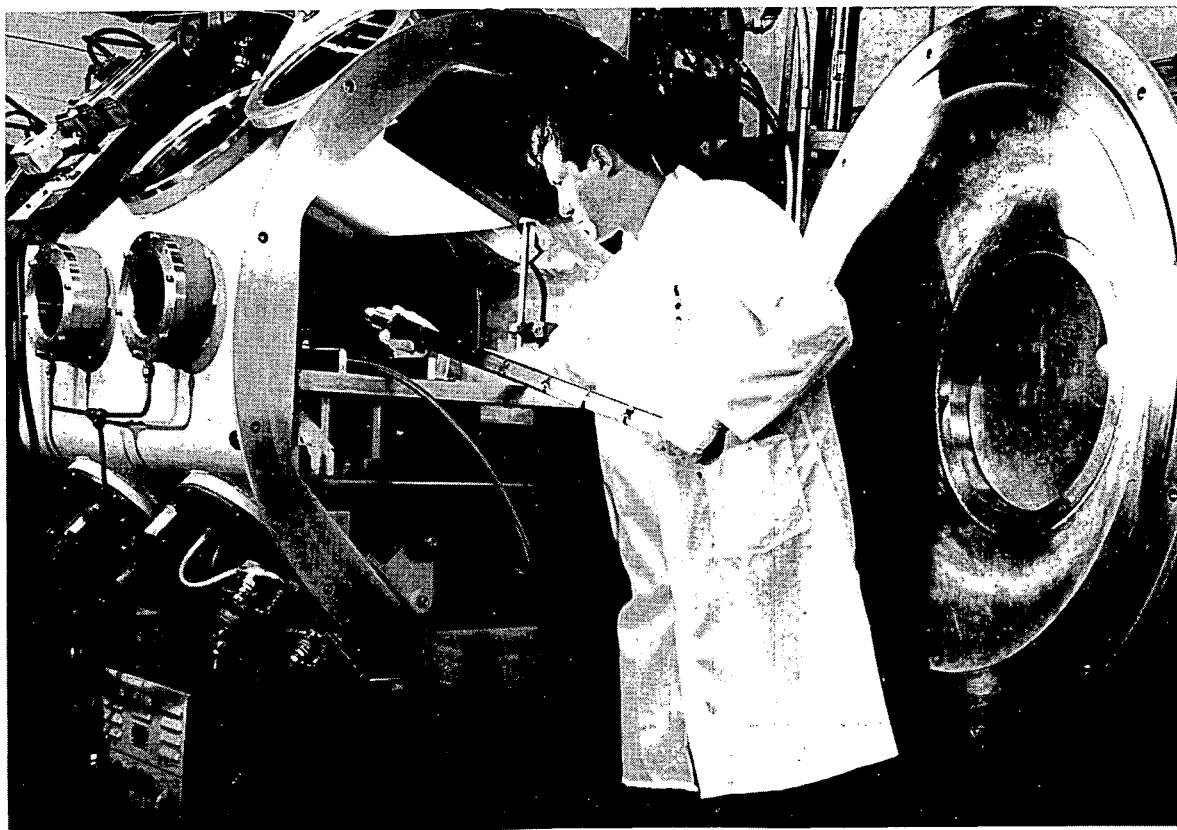


Figure IX-7. - Vacuum purged weld chamber.

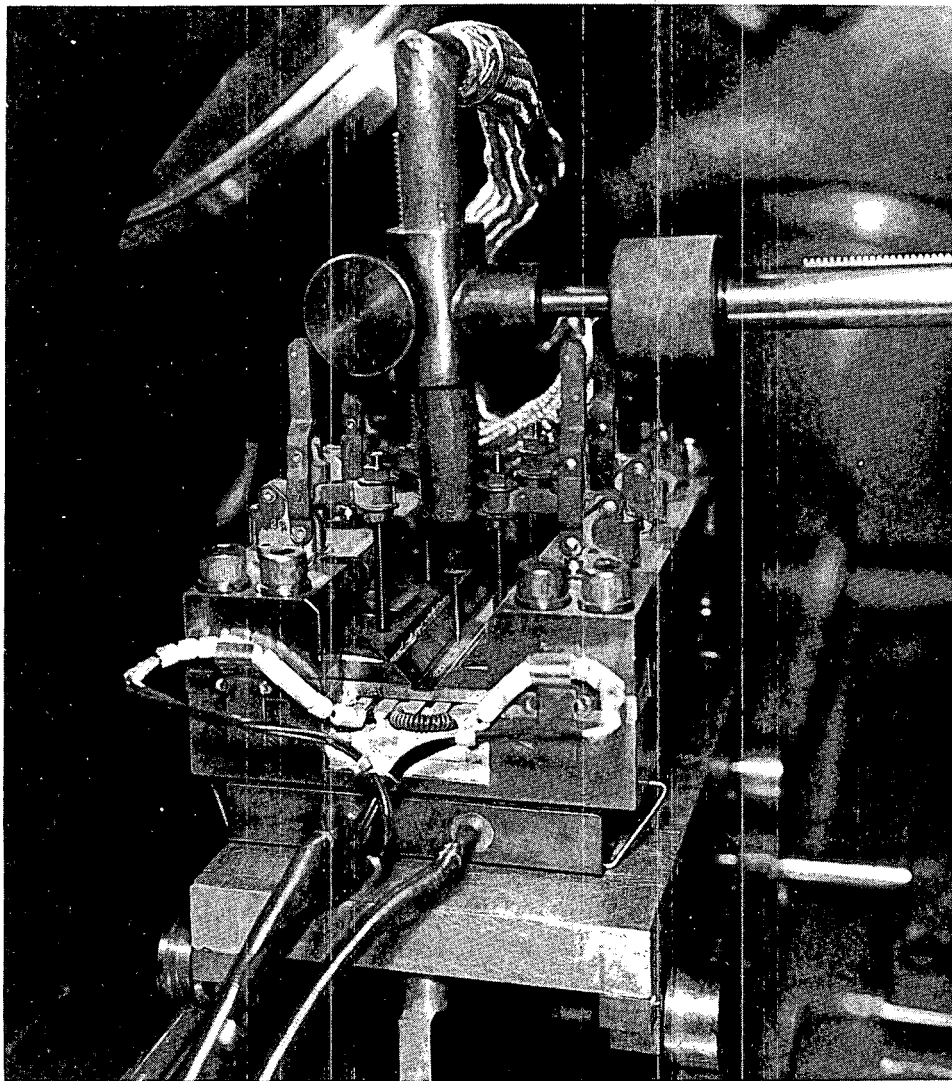


Figure IX-8. - Sheet welding fixture used for welding tungsten-base alloys with preheat to 1400° F.

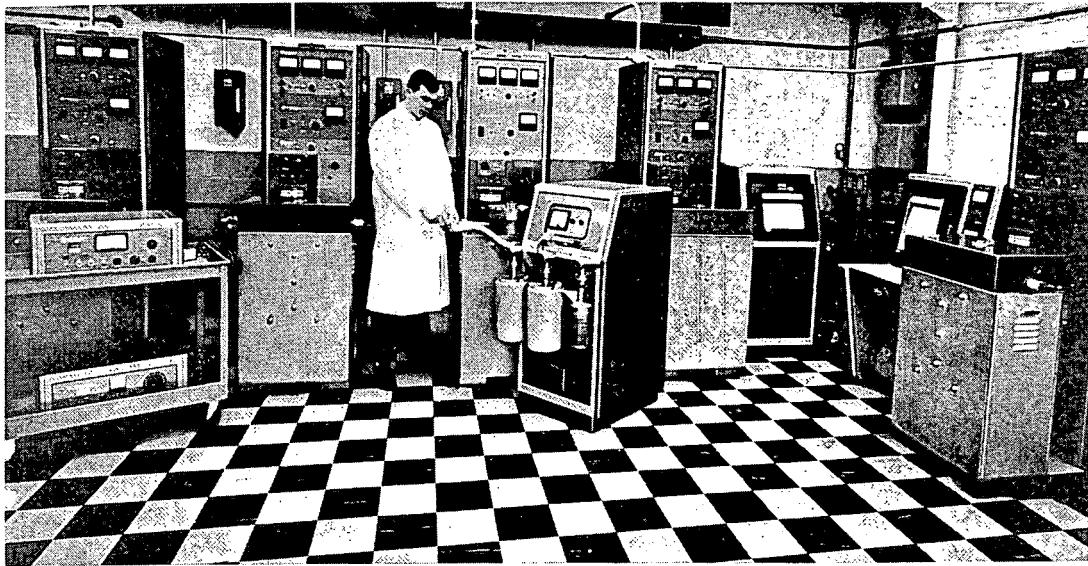


Figure IX-9. - Sputter ion pumped ultrahigh vacuum furnaces used for thermal stability study.

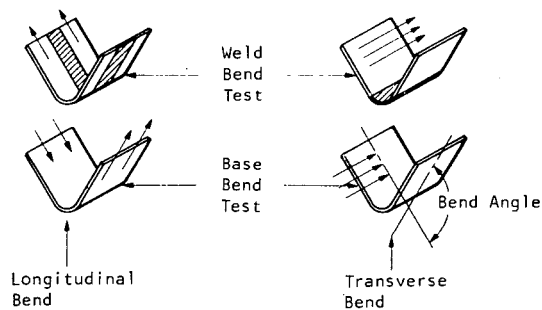


Figure IX-10. - Bend test specimens. Thickness,  $t$ , 0.035 inch; width,  $12t$ ; length,  $24t$ ; test span,  $15t$ ; punch speed, 1 inch per minute; variable temperature; variable punch radius, generally  $1t$ ,  $2t$ ,  $4t$ , or  $6t$ ; bend ductile to brittle transition temperature, lowest temperature for  $90^\circ$  bend without cracking. Arrows show rolling direction.

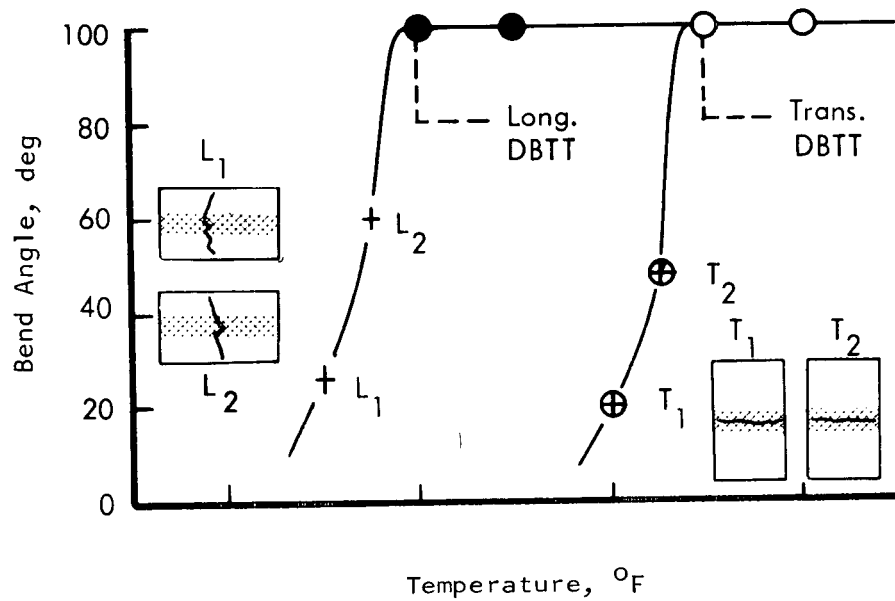
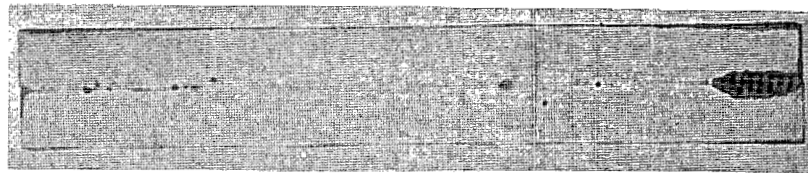


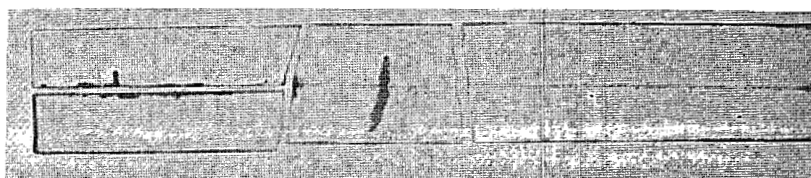
Figure IX-11. - Method of recording bend test data for analysis (GTA welds shown).



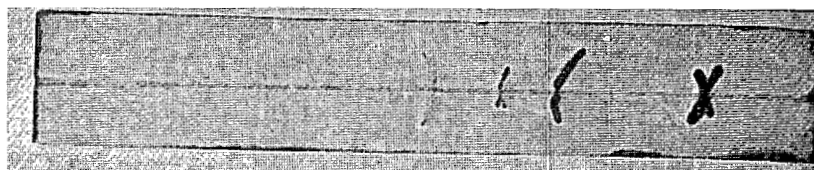
Weld 1; speed, 15 in. /min; 2.98 kJ/in.



Weld 4; speed, 15 in. /min; 3.24 kJ/in.



Weld 7; speed, 50 in. /min; 1.19 kJ/in.



Weld 10; speed, 15 in. /min; 3.12 kJ/in.



Weld 11; speed, 50 in. /min; 1.30 kJ/in.



Weld 12; speed, 100 in. /min; 0.76 kJ/in.

Figure IX-12. - Typical dye-penetrant results of electron beam welds in arc cast unalloyed tungsten sheet.



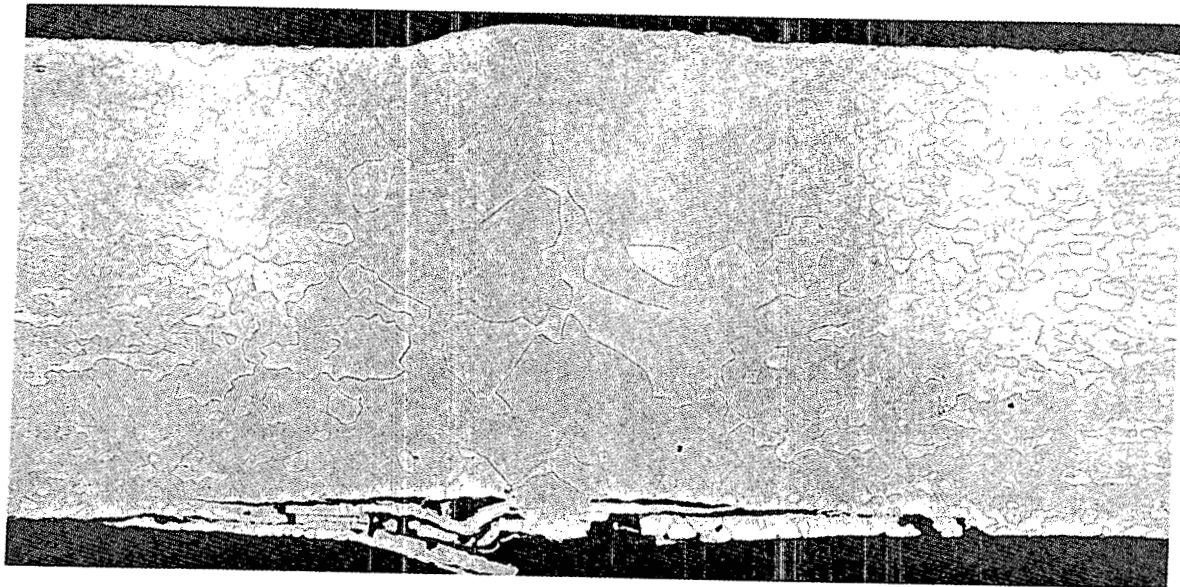


Figure IX-13. - Typical section of electron beam weld in unalloyed tungsten.  $\times 80$ .

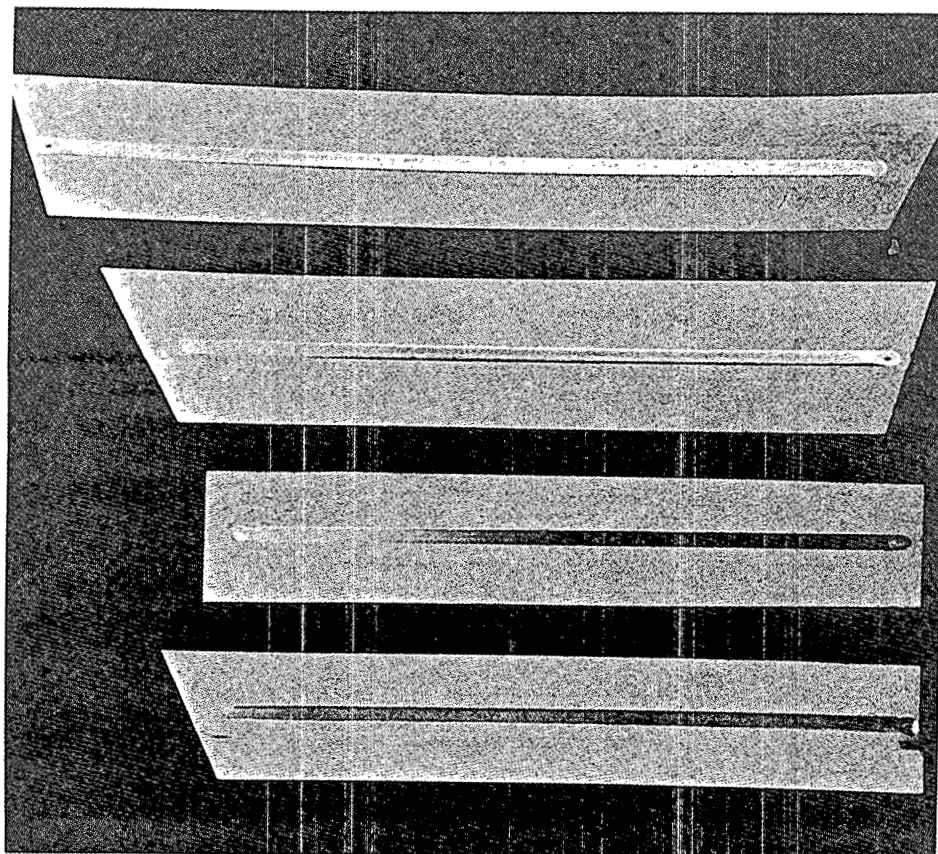


Figure IX-14. - Bead-on-plate GTA welds on 0.030-inch powder metallurgy W-25Re-30Mo alloy sheet.

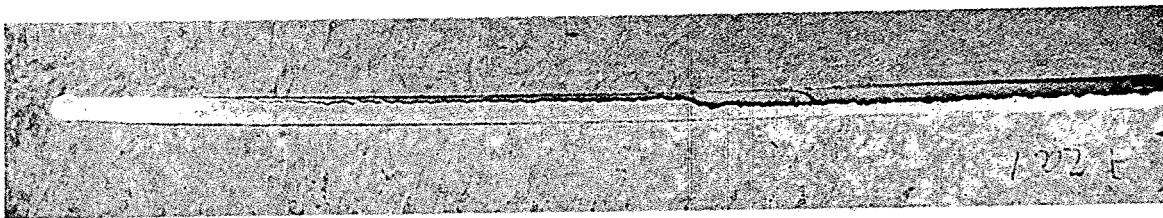
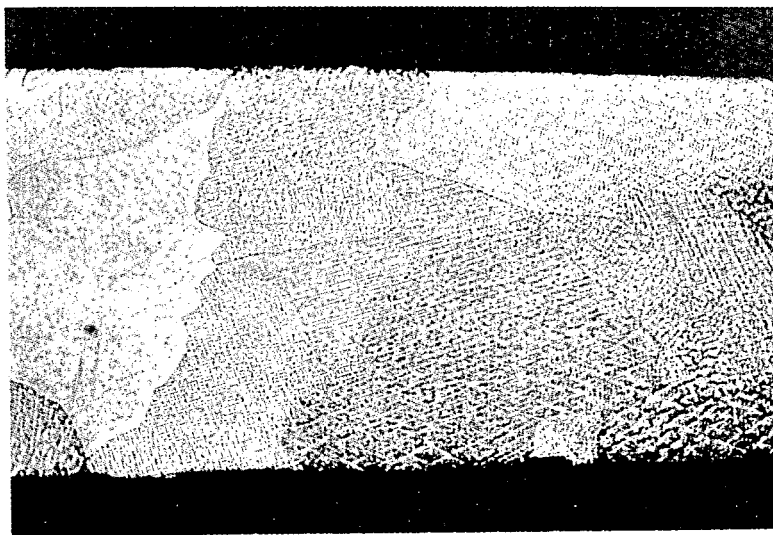


Figure IX-15. - Typical hot tear on bead-on-plate GTA weld on 0.030-inch arc cast W-25Re-30Mo alloy sheet.

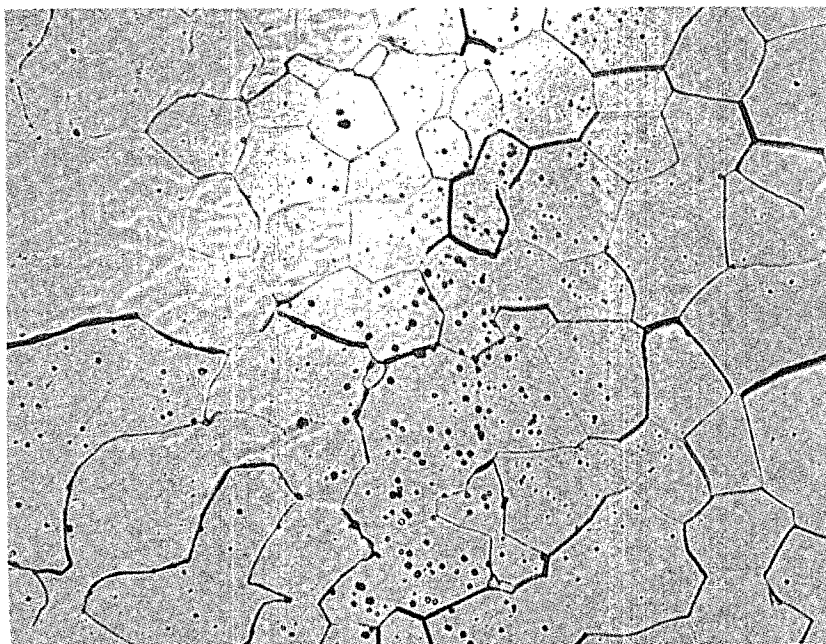


Prepared using 30 lactic-3HNO<sub>3</sub>-1HF (vol. ratio)

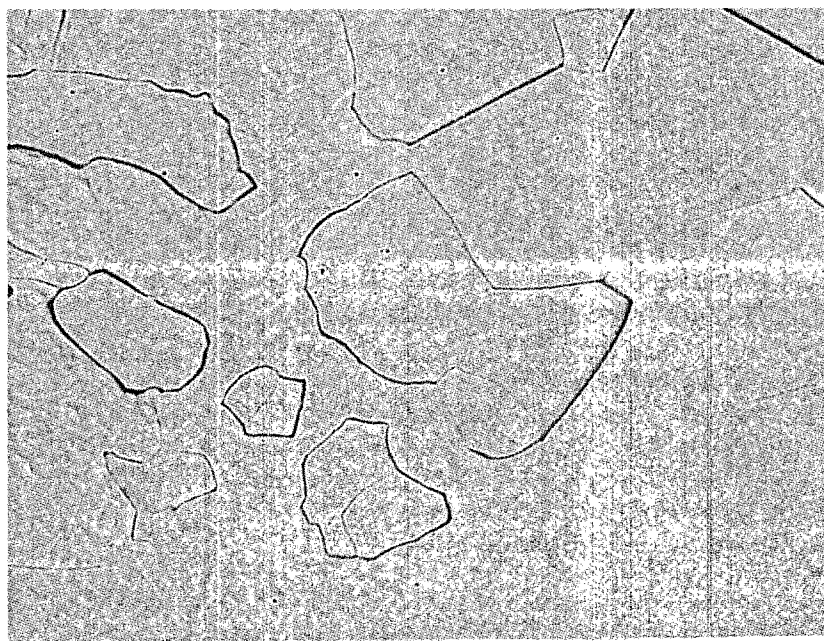


Prepared using 9HF-1HNO<sub>3</sub> (vol. ratio)

Figure IX-16. - Center areas of GTA welds in W-25Re-30Mo sheet showing effect of pickling solution used for joint preparation.  $\times 75$ .

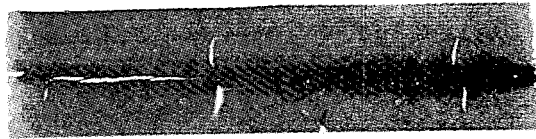
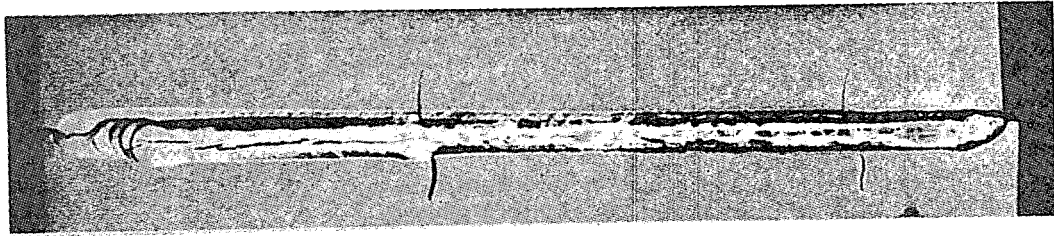


Powder metallurgy

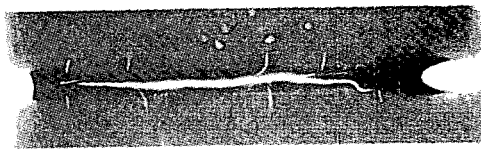
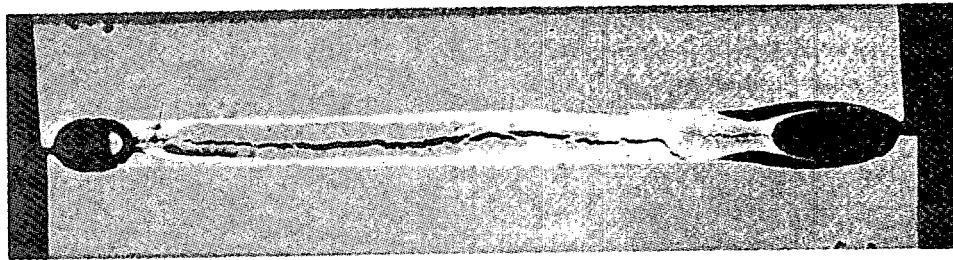


Arc cast

Figure IX-17. - Comparison of typical porosity levels in GTA HAZ welds in powder metallurgy and arc cast W-25Re-30Mo alloy sheet.  $\times 400$ .



500 ppm Oxygen in Weld Atmosphere



1800 ppm Oxygen in Weld Atmosphere

Figure IX-18. - GTA Welds in PM W-25Re-30Mo sheet. Weld atmospheres contaminated with oxygen as indicated.

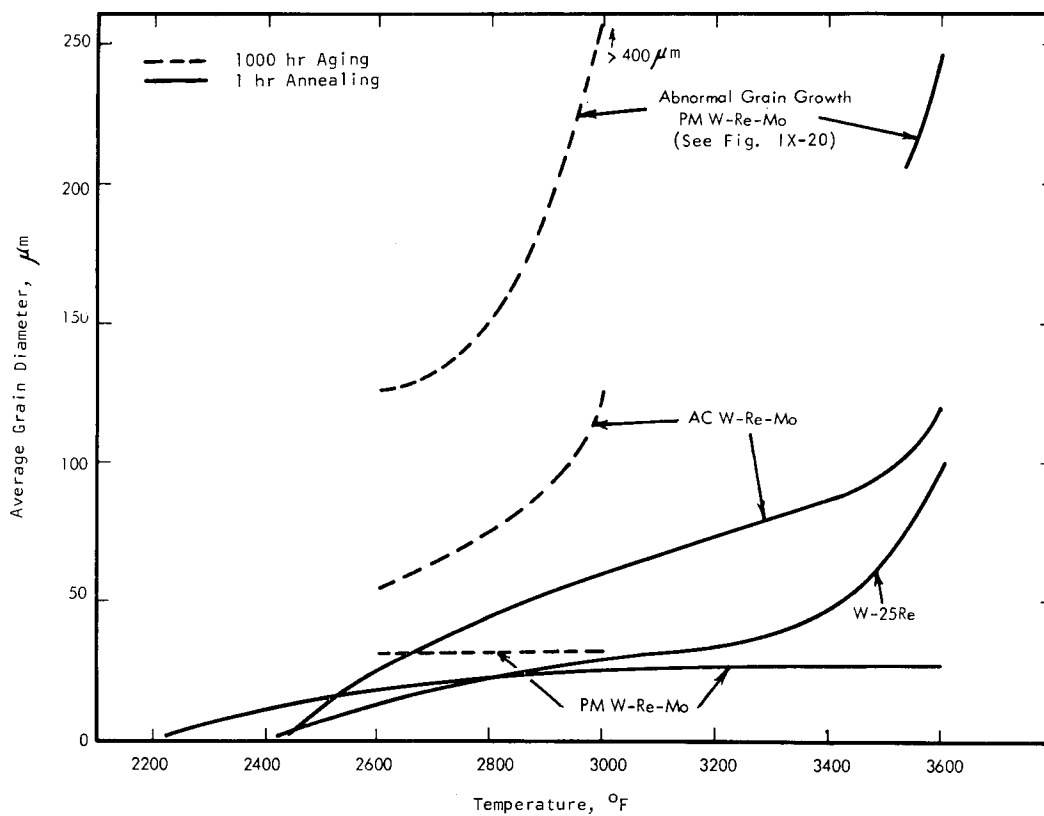
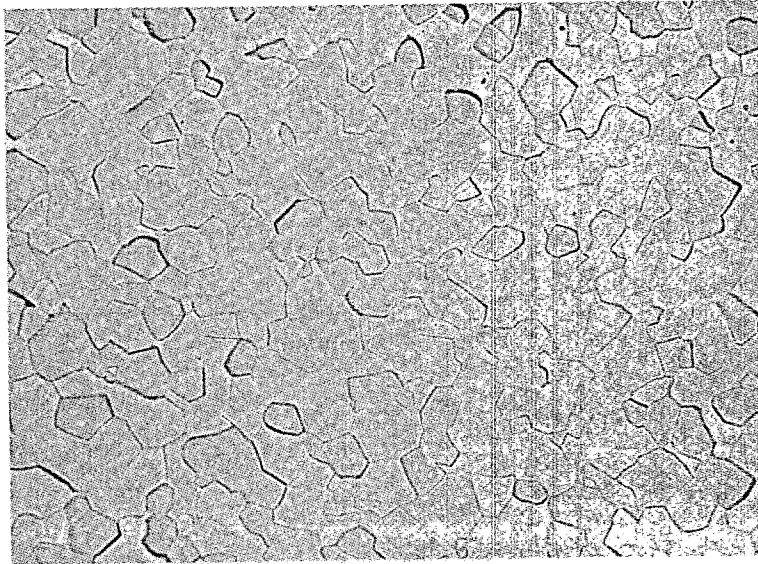
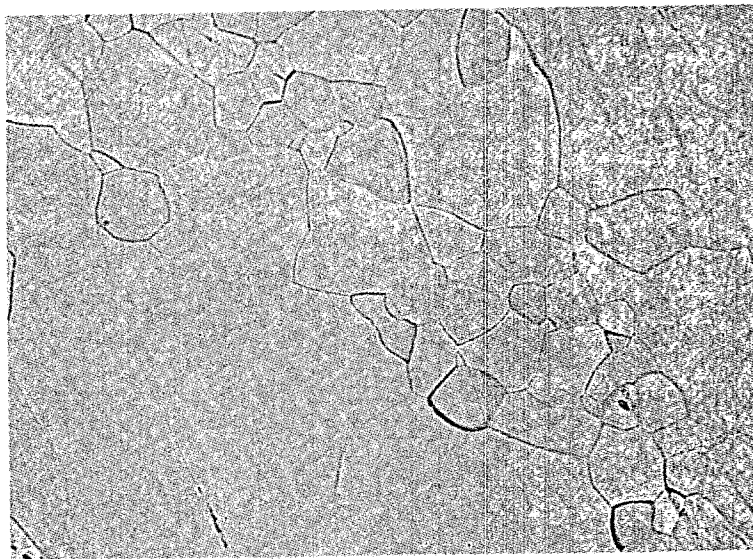


Figure IX-19. - Average grain size as function of 1000 hour aging and 1 hour annealing temperature for tungsten-base alloys.





3400° F



3600° F

Figure IX-20. - Microstructure of powder metallurgy W-25Re-30Mo sheet following 1 hour anneals at temperatures indicated. Abnormal grain growth occurred at 3600° F.  $\times 200$ .